Laser Peening Study for Reduction in Hydrogen Permeation in a Titanium Alloy

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This project investigates the permeation of hydrogen into metals and alloys. Hydrogen-rich environments, such as fuel cell reactors, can exhibit damage caused by hydrogen permeation in the form of corrosion cracking, by lowering tensile strength and decreasing material ductility. This can lead to corrosion cracking or material failure.

Coatings and liners have been investigated but there are few shot-peening or laser-peening studies on preventing hydrogen embrittlement. The surface compressive residual stress induced by laser peening is successful in preventing stress corrosion cracking for stainless steels in power plants. The question is whether the residual stresses induced by laser peening can delay or enhance the penetration of hydrogen into a material.

Project Goals

Three areas of study are required: laser peening the material, hydrogenation, and hydrogen detection. If laser peening proves successful in delaying hydrogen embrittlement of material, it could aid in extending the life of many types of systems. Equipment components used in the production of these hydrogen isotopes often have finite service lives due to degradation by hydrogen penetration. Extending the lifetime would potentially have significant cost savings as well as implications for higher performance. Hydrogen embrittlement also will have significant impact as the United States moves to a hydrogen-based fuel economy. This process could potentially improve fuel cell performance and could allow for safer and denser storage of hydrogen.

Relevance to LLNL Mission

Of particular interest to the Laboratory and DOE is the potential benefit of this study for applications in deuterium and tritium production and storage. The process could be used to extend the life of components that store hydrogen and its isotopes, and to a broad range of metals subjected to corrosive environments. Such critical parts include gas storage vessels for nuclear weapons, the tools and machines used to form these components, equipment used to produce the gas, and nuclear storage containers.

FY2004 Accomplishments and Results

For our material preparation and hydrogen detection work, we tested Ti-6Al-4V double phase. One coupon was laser peened with 10 GW/cm^2, 18-ns pulse width, and two layers of peening.

Cathode charging was performed on the as-received and laser-peened specimens. Each specimen was fully immersed in a vessel of a four-port electrochemical cell containing an aqueous electrolytic charging solution of 5% H_2SO_4 at ambient temperature. A precision power supply was connected by clip leads to the three electrodes and provided a constant, uniform exchange

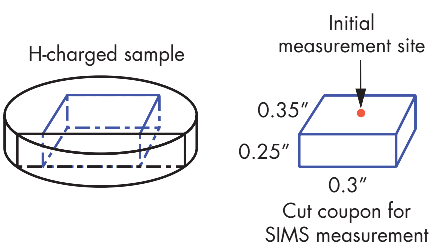

Figure 1. Initial hydrogen-charged coupon and SIMS measurement location.

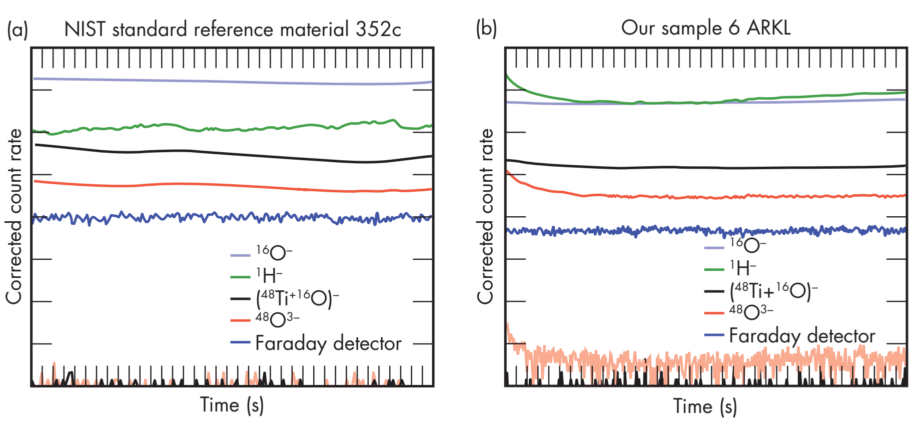

Figure 2. NIST Standard reference material (a) and our sample (b).

current density of 10 mA/cm^2 onto the specimen, over a charging duration of 144 h.

The parameters for the secondary ion mass spectroscopy (SIMS) measurement are as follows: a pattern 250 μm × 250 μm in size was rastered across the surface of the coupon, to a depth of 85 μm. Figure 1 shows the coupon dimensions and initial measurement location. The ions collected from this trace were compared with a standard reference material (352 c) containing hydrogen concentration of 49.0± 0.9 μg/g in unalloyed titanium. The trace made on the NIST standard material was assumed to be homogeneous (see Fig. 2).

Four main ions were recorded in each sample, $^{16}O^{-}$, $^{1}H^{1-}$, $(^{48}Ti^{+16}O)^{-}$, and O^3. The scans of each coupon showed very consistent levels of each ion to the depth measured. Results also show the counts of each ion to be very constant over each scanned volume. This means the material is fairly uniform in the region measured, allowing a fair comparison of each ion concentration between the two samples.

The data in Fig. 3 show the recorded ion counts for each ion measured. When compared to the standard, our sample showed almost ten times the ratio of H^{1-} ions as compared to the standard, or roughly 490 μg/g. This demonstrates that we can measure the hydrogen content in our Ti-6Al-4V material and quantify its concentration. Since we want to compare the hydrogen embedding depth in LP versus non-LP samples, our next step is to measure three coupons with various treatments using a step scan method.

Related References

1. Peyre, P., C. Braham, J. Ledion, L. Berthe, and R. Fabbro, *Journal of Materials Engineering and Performance*, **9**, pp. 656-662, 2000.
2. Peyre, P., X. Scherpereel, L. Berthe, C. Carboni, R. Fabbro, G. Beranger, and C. Lemaitre, *Material Science and Engineering*, **A280**, pp. 294-302, 2000.
3. Iost, A., and J. B. Vogt, *Scripta Metallurgical et Materialia*, **37**, pp. 1499-1504, 1997.

FY2005 Proposed Work

We would like to investigate the hydrogen charging conditions to optimize the parameters for hydrogen penetration. Investigating different cathode charging conditions, such as current density and charging time, would allow for a more accurate comparison of hydrogen penetration environments.

NIST Standard with 49.0µg/g Hydrogen Concentration

Block data for DR4722A.002 (SRM 352c initial primary DVM reading: 0.27862)
Raster ON

Block data parameters:
Using Ti+O as reference
Starting cycle: 1 Ending cycle: 190 Cycles per block: 190

	Block Isotope Ratios							
	Mass/ Ti+O	Mean ratio	Minimum ratio	Maximum ratio	DX/X (O/O)	Sigma (O/OO)	Sigma /sqr	Sigma mean
	1.000	1.3901E–06	0.0000E+00	7.6289E–06	548.8	1285.9	1.0	1.3002E–07
	1.001	0.03356930	0.01295870	0.06272485	148.2	284.5	38.6	0.00069478
$^1H^-$ Ion	^1H	4.68946743	1.90224540	9.01404762	151.7	393.2	225.6	0.13411221
	^{16}O	5.3497E+01	3.7079E+01	7.0225E+01	62.0	174.2	105.7	0.67799244
	O+O+O	0.17235045	0.13389201	0.20850052	43.3	110.8	20.1	0.00138951
	62.6	1.6766E–06	0.0000E+00	1.0675E–05	636.7	1338.7	1.1	1.6327E–07
Reference ion $(^{48}Ti^{+16}O)^-$	Ti+O	1.00002456	0.98006779	1.01774096	3.8	3.7	1.6	0.00027026

H-Charged Sample

Block data for DR4723A.001 (6 AR KL laser-peen initial primary DVM reading: 0.27763)
Raster ON, 10E+10 ohm on FC

Block data parameters:
Using Ti+O as reference
Starting cycle: 1 Ending cycle: 500 Cycles per block: 500

	Block Isotope Ratios							
	Mass/ Ti+O	Mean ratio	Minimum ratio	Maximum ratio	DX/X (O/O)	Sigma (O/OO)	Sigma /sqr	Sigma mean
	1.000	2.9703E–06	0.0000E+00	2.8516E–05	960.1	1340.5	1.1	1.7824E–07
	1.001	0.03082788	0.01911497	0.04381545	80.1	146.1	13.8	0.00020156
$^1H^-$ Ion	^1H	4.3765E+01	3.3135E+01	1.1504E+02	187.5	230.0	124.5	0.44969863
	^{16}O	3.3581E+01	2.3614E+01	3.8100E+01	43.1	104.0	55.3	0.15639845
	O+O+O	0.22053018	0.17884158	0.56418800	174.7	221.5	38.6	0.00218714
	62.6	2.8683E–05	0.0000E+00	1.3040E–04	454.6	524.6	1.3	6.7366E–07
Reference ion $(^{48}Ti^{+16}O)^-$	Ti+O	1.00002277	0.98597926	1.02062881	3.5	4.4	1.7	0.00019492

Figure 3. Measurement results for ion concentration.